Determination of the d-Limonene Content in Orange Rind Using gas Chromatography

Lab Report # 6

4/26/2010

Chem 216

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# Purpose:

The purpose of this experiment is to determine the amount of *d*-limonene present in the orange rind by applying the gas chromatographic techniques, using the HP/Agilent 4890 chromatograph with the FID detector.

# Procedure:

Refer to the lab manual. While neglecting to prepare the standard solution since they were already prepared by the TAs before our entry to the lab.

# Data:

The data was collected from the group of Jad Houssami and Elie Abdelnour

Mass of anisole added to extract=0.0350g

Mass of rind = 10.0290g

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Trial 1** | **mA** | **mL** | **mL pure added** | **Aa** | **AL** |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 572 | 517 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 3200 | 5682 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2730 | 6971 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 5319 | 23074 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2825 | 13832 |
| Dilute Unknown |  |  |  | 311382 |
| Concentrated Unknown |  | 110009 | 929790 |
|  |  |  |  |  |  |
| Trial 2 | mA | mL |  | Aa | AL |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 2581 | 1603 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 1908 | 3510 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2997 | 9322 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 4217 | 17263 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2852 | 6508 |
| Concentrated Unknown |  | 6768 | 53815 |
|  |  |  |  |  |  |
| Average | mA | mL |  | Aa | AL |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 1576.5 | 1060 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 2554 | 4596 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2863.5 | 8146.5 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 4768 | 20168.5 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2838.5 | 10170 |
| Concentrated Unknown |  | 58388.5 | 491802.5 |

# Calculations:

ML/MA= mass of Limonene divided (95% pure Limonene sample) by mass of anisole (the internal standard)

aA/aL= GC peak area of Limonene divided by mass of GC peak area of anisole

Peak Area of Limonene/ Peak Area of anisole = AL/Aa

ML pure= ML$×0.95$

## Sample calculation:

Standard 1:$ \frac{1060}{1576.5}=$0.672375515

Standard 1: ML pure= 0.0624$×0.95$ = 0.05928

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Trial 1** | **mA** | **mL** | **mL pure added** | **Aa** | **AL** | **mL/mA** | **AL/Aa** |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 572 | 517 | 0.522291 | 0.903846154 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 3200 | 5682 | 0.984846 | 1.775625 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2730 | 6971 | 1.396791 | 2.553479853 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 5319 | 23074 | 1.795455 | 4.338033465 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2825 | 13832 | 2.277071 | 4.896283186 |
| Dilute Unknown |  |  |  |  | 311382 |  |  |
| Concentrated Unknown |  |  |  | 110009 | 929790 |  | 8.451944841 |
|  |  |  |  |  |  |  |  |
| Trial 2 | mA | mL |  | Aa | AL | mL/mA | AL/Aa |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 2581 | 1603 | 0.522291 | 0.621077102 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 1908 | 3510 | 0.984846 | 1.839622642 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2997 | 9322 | 1.396791 | 3.110443777 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 4217 | 17263 | 1.795455 | 4.093668485 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2852 | 6508 | 2.277071 | 2.281907433 |
| Concentrated Unknown |  |  |  | 6768 | 53815 |  | 7.951388889 |
|  |  |  |  |  |  |  |  |
| Average | mA | mL |  | Aa | AL | mL/mA | AL/Aa |
| Std 1 | 0.1135 | 0.0624 | 0.05928 | 1576.5 | 1060 | 0.522291 | 0.672375515 |
| Std 2 | 0.1036 | 0.1074 | 0.10203 | 2554 | 4596 | 0.984846 | 1.799530149 |
| Std 3 | 0.1044 | 0.1535 | 0.145825 | 2863.5 | 8146.5 | 1.396791 | 2.844944997 |
| Std 4 | 0.1045 | 0.1975 | 0.187625 | 4768 | 20168.5 | 1.795455 | 4.229970638 |
| Std 5 | 0.1038 | 0.2488 | 0.23636 | 2838.5 | 10170 | 2.277071 | 3.582878281 |
| Concentrated Unknown |  |  |  | 58388.5 | 491802.5 |  | 8.422934311 |

## Calibration curve

And the calibration curve of AL/Aa vs mL/mA is:

By interpolation, we chose Y=8.422934311, we find X=3.360 from the equation of the calibration curve.

X is the mass ratio of limonene to anisole. ML/MA= 3.360

Hence, ML= 0.035x 3.360=0.1176 g

In 10 grams of Rind, %Limonene = (ML/Mrind)x 100 = 0.1176/10.0290x100 = 1.173%

## Error in calculation:

ML/MA=2.7622× (aL/aA)-0.8585

ML=(2.7622× (aL/aA)-0.8585 )MA

Uncertainty in weighing the Anisole= $\sqrt{2(0.0001)^{2} }=0.$**00014**

Standard deviation in the (aL/aA) calculation from the two repetitions= $\genfrac{}{}{0pt}{}{\sqrt{\sum\_{}^{}(x-xav)^{2}}}{}$=0.354

Relative Error on the Mass of Limonene= $\sqrt{(\frac{0.00014}{0.035})^{2}+(\frac{0.354}{8.422934311})^{2}= } $ 0.042

Error on the mass of Limonene = 0.042 $×$0.1176 g = 0.005

Relative Error on the percentage mass of Limonene = $\sqrt{(\frac{0.005}{0.1176})^{2}+(\frac{0.00014}{10.029})^{2} }$ =0.043

Error on the percentage mass of Limonene = 0.043 x 1.173 = 0.05

# Results:

Mass of Limonene = 0.1176 g

% of Limonene = 1.173%

# Discussion:

 There were no difficulties in collecting our data, everything was clear in the graphs both Limonene and Anisole were clearly present and all the required values were found. The advantages of the internal standard method were that it was an easy procedure and it gave us a relationship which allows us to reach the required objective.